doi: 10.1017/S1431927609099218
Microsc Microanal 15(Suppl 2), 2009

A High Temperature ESEM Investigation Into The Separation Of $MgCl_2$ From $MgCl_2$ – Ti Mixture.

A. M. Glenn*, C. Doblin**, C. M. MacRae*.

* Microbeam Laboratory, CSIRO Minerals, Box 312, Clayton South, VIC, 3169, Australia.
** CSIRO Minerals, Box 312, Clayton South, VIC, 3169, Australia.

matthew.glenn@csiro.au

The most important method for the commercial production of titanium is the Kroll process, invented in 1940. This is a batch process which produces titanium “sponge” from the reduction of $TiCl_4$ by magnesium powder according to the following reaction [1]:

$$TiCl_4 + 2Mg \rightarrow Ti + 2MgCl_2$$

The Commonwealth Scientific and Industrial Research Organisation (CSIRO) have developed a novel, proprietary process, TiRO™, to reduce the overall cost of producing titanium powder [2, 3]. Instead of the batch system, TiRO™ uses a continuous, argon gas, fluidised bed reactor to produce the Ti – $MgCl_2$ mixture in the form of discrete particles. A typical example of these particles is shown in Figure 1. In a second step, the $MgCl_2$ is separated from the titanium by volatilisation in a vacuum distillation furnace. This results in a relatively pure, friable matte of fine titanium particles. The particles adopt either a honeycomb or shell like morphology as shown in Figure 2. However, the mechanisms by which the titanium matte is formed have not been directly observed and so are not yet fully understood [4]. Therefore, the objective of this investigation was to directly observe the volatilisation of $MgCl_2$ in real time by simulating the vacuum distillation process inside the environmental scanning electron microscope using its hot stage and controlled atmosphere.

Initial calculations suggested that oxygen levels in the chamber should be in the vicinity of 10 parts per million to prevent premature oxidation of the titanium. Therefore, after installing the hot stage and placing a very small amount of the $MgCl_2$ – Ti mixture into its crucible the following precautions were taken to minimise oxygen levels: overnight pumping before the experiment, use of an ultra high purity argon atmosphere, use of a liquid nitrogen cold trap to further improve the vacuum, complete removal of the water vapour supply line to the chamber and use of some sacrificial titanium powder sprinkled around the sample to simulate the action of an ion getter pump. Furthermore, a residual gas analyser (RGA) was installed to measure the oxygen content of the chamber. The RGA measurements, Figure 3, suggested that overnight pumping reduced the oxygen and water vapour partial pressures by an order of magnitude. However, energy dispersive x-ray spectrometry (EDS) performed on the titanium after completion of the experiment, shown in Figure 4, suggested that the oxygen content was still not low enough and that additional oxygen removal strategies will be required. These may include use of a titanium oxygen getter in the argon line or use of a carbon monoxide atmosphere to further reduce the oxygen partial pressure. In addition, the EDS results, Figure 4, indicate considerable quantities of magnesium chloride remain in the sample after the experiment suggesting that the temperature attained by the sample in the hot stage was not high enough for the volatilisation reaction to proceed. Measures to improve the heat flow into the sample, such as further insulation and removal of thermal resistance, will be undertaken to enhance chloride removal rates.

References

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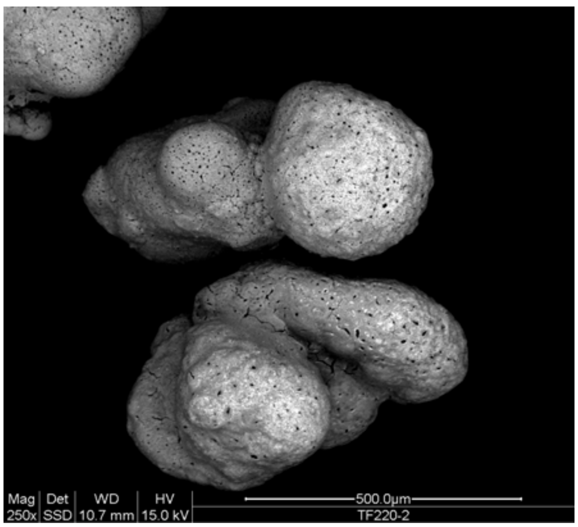

Fig. 1. Backscattered electron image showing discrete particles of $MgCl_2$ – Ti mixture.

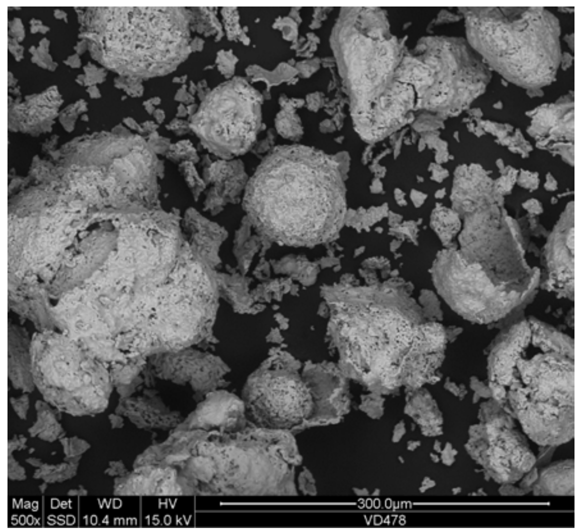

Fig. 2. Backscattered electron image of titanium particles after vacuum distillation.

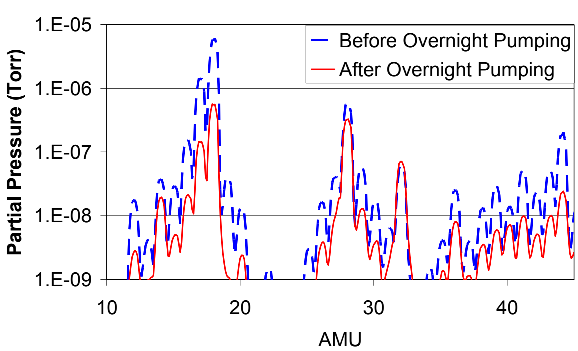

Fig. 3. RGA results showing partial pressures of oxygen and water vapour measured before and after overnight pumping.

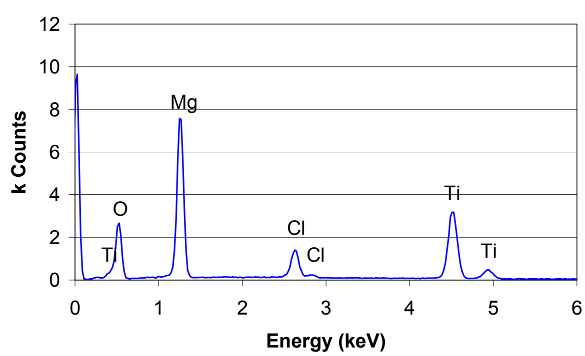

Fig. 4. EDS results of Ti – $MgCl_2$ particle after experiment has been completed.

https://doi.org/10.1017/S1431927609099218 Published online by Cambridge University Press